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Journal of Alloys and Compounds

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Letter

Enhancing the coercivity, thermal stability and exchange coupling of nano-composite (Nd,Dy,Y)–Fe–B alloys with reduced Dy content by Zr addition



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ARTICLE INFO

Article history: Received 26 November 2013 Received in revised form 1 April 2014 Accepted 1 April 2014 Available online 13 April 2014

Keywords: Rare earth hard magnets Nanocomposite alloys Melt-spinning Thermal stability

ABSTRACT

With the intention to reduce Dy content in NdFeB based magnets, 50 at.% Y substituting Dy was previously successfully employed to improve the remanence and thermal stability of the nanocomposite $[Nd_{0.8}Dy_{0.2}]_{10}Fe_{84}B_6$ alloy without the energy product reduction. In this work, introducing Zr into Y substituted alloys has enhanced the coercivity H_{cj} of the melt spun $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}Fe_{84-x}B_6Z\Gamma_x$ alloys. With increasing x value from 0 to 2, H_{cj} increased from 575 to 814 kA/m. Doping 2 at.% Zr reduced the absolute value of the temperature coefficient β from 0.394 to 0.348%/°C. Good magnetic properties with H_{cj} of 797 kA/m, maximum energy product $(BH)_{max}$ of 131 kJ/m³ and β of -0.356%/°C were obtained for x = 1.5. Both the Curie temperature and lattice constants of the hard magnetic phase decreased with Zr addition, indicating that Zr atoms can substitute directly into the hard phase, although some atoms may also locate outside the lattice. Together with the analysis on the demagnetization curve and recoil loops, the results verified that a small amount of Zr can improve the coercivity, thermal stability and exchange coupling of nanocomposite NdDyYFeB alloys through enhancing the anisotropy and improving the microstructure.

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1. Introduction

NdFeB based alloys have been employed as the high performance permanent magnets for almost thirty years. For the sintered NdFeB magnets to be used at relatively high temperatures, the heavy rare earth (RE) element Dy has been frequently added to improve the elevated temperature behavior by enhancing the coercivity H_{cj} . However, it becomes a major concern that Dy addition brings up the price of the magnets [1,2]. In order to reduce Dy content for NdFeB magnets, various approaches including grain refinement and grain boundary diffusion processes have been successfully developed [3]. Elemental substitution and addition have also been tried earlier but were not quite successful [4]. New alloy design, therefore, is urgently in need. For bonded or die-upset magnets based on melt spun nanocrystalline alloy ribbons, improving the properties/cost ratio is also a major concern. In our previous

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work [5], the advantages of Y substituting Dy for the thermal stability of $(NdDy)_2Fe_{14}B/\alpha$ -Fe alloys have been investigated. Fe-rich composition was selected since this type of magnets has shown enhanced remanent polarization (remanence) J_r and maximum energy product $(BH)_{max}$ as well as low temperature coefficients due to the exchange-coupling between magnetically hard and soft phases [6]. Improved J_r and thermals stability have been obtained in the nanocomposite $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}Fe_{84}B_6$ alloy. The enhanced properties result from the higher saturation magnetization and the more weak temperature dependence of coercivity for Y₂Fe₁₄B compound than for Dy₂Fe₁₄B [7]. Although a similar value of $(BH)_{max}$ as Y free alloy was obtained in this composition, H_{ci} for above mentioned alloy is only 575 kA/m, which is much lower than the single phase Nd₂Fe₁₄B alloy. Hence, enhancing the coercivity of the Y substituted alloy is very important for achieving high performance in NdFeB alloys with low Dy content.

The magnetic properties of nanocomposite magnets are closely dependent on their microstructure. Uniform fine grain structure is beneficial to the exchange interaction and magnetic performance. The element doping has been frequently employed to improve the microstructure and enhance the coercivity of the NdFeB alloys.

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The role of doping elements like Ti, Ga, Zr, Nb, etc. in NdFeB magnet have been reported before [8-11]. Among these elements, the effects of Zr are still not quite clear, although it has been practically employed in sintered magnets for enhancing the coercivity. Capehart et al. [12] suggested that Zr atoms get into the crystal lattice of Nd₂Fe₁₄B phase and occupy the positions of Nd atoms. Jurczyk and Wallace [13] found that Zr substituting RE led to the reduced lattice constants and Curie temperature, but increased anisotropy field. However, Fidler and Schrefl [14] claim that Zr is intended to form binary ZrRE2 phase in NdFeB alloys. Raviprasad et al. [15] found the Zr-containing compound in the grain boundary, which cannot pin the domain wall and, instead, they can be the nucleation center for reversal domain and lead to reduced coercivity. Therefore, the role of Zr in NdFeB alloys is still in need of further research. Except that, until now, the elevated temperature behavior of the NdFeB alloys with both Y substitution and Zr doping has seldom studied. The effects of Y and Zr on the magnetic properties have to be further clarified.

This paper reported our recent work on the Zr doping of the Y substituted nanocomposite NdDyFeB alloys. The magnetization reversal behavior and exchange coupling for these alloys are also investigated. The obtained results provide new guidance for the design of nanocrystalline NdFeB alloys.

2. Experimental

Nanocomposite $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}Fe_{84-x}B_6Zr_x~(x=0-5)$ alloys were prepared by argon arc melting followed by melt spinning. Raw materials of Nd, Dy, Y, Fe, Fe–B and Zr with purity higher than 99.8% were arc melted to produce small buttons under Ar atmosphere. The buttons were melted for six times to ensure the compositional homogeneity. All examined as-spun ribbons were produced at an optimized wheel speeds varied between 7 and 20 m/s depending on the composition. The phase constitution was characterized by X-ray diffraction (XRD, Philip X-pert) using Cu Kα radiation. The magnetic properties of directly quenched alloys were tested by physical property measurement system (PPMS, Quantum Design Co., USA) equipped with a vibrating sample magnetometer (VSM) using a maximum magnetic field of 8 T. Magnetization–temperature (M–T) curves were measured at a magnetic field H = 1000 Oe between 300 and 1000 K.

3. Results and discussion

By optimized melt spinning process, the experimental alloys with nanocrystalline structure have been obtained. The typical microstructure of the alloys are shown in Fig. 1, obtain from $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}Fe_{82.5}B_6Zr_{1.5}$ melt spun ribbon. The nanocomposite structure has been demonstrated. The grain sizes of hard and soft phases are between 20 and 50 nm. No significant change was found for various compositions.

3.1. Effect of Zr on the magnetic properties

Fig. 2 shows the hysteresis loops for $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}$ $Fe_{84-x}B_6Zr_x$ alloys with various Zr doping. For x = 0-2, all alloys have good loop squareness with single phase characteristics, indicating well exchange coupled hard and soft magnetic grains. The squareness factor S^* , shown in Fig. 3, is defined by the ratio of the reverse field required to reduce J by 10% from the remanence to H_{cj} . The value for the alloy with x = 5 is not shown since an obvious lost of squareness can be found in Fig. 2. There are several other methods to quantify the squareness of the loop, such as the ratio of I_r-I_s , which is also shown in Fig. 3 for the experimental alloys. Although I_r/I_s has no significant change, the squareness factor S^* is improved by increasing Zr content from x = 0 to 1.0. The enhanced squareness is possibly due to the improved uniformity of the microstructure [16], which has also been demonstrated by the magnetic analysis, as discussed later. For x = 3 and 5, the loop squareness became worse, indicating some changes in the microstructure. With the nonmagnetic Zr doping, the saturation

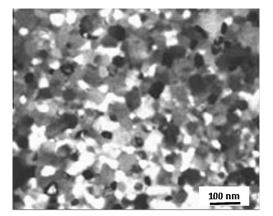


Fig. 1. The typical microstructure of the alloys are shown in this figure, obtain from $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}Fe_{82.5}B_6Zr_{1.5}$ melt spun ribbon.

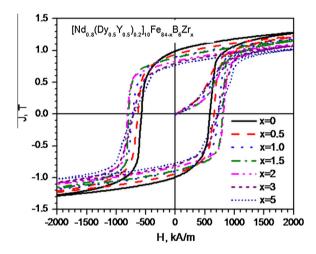


Fig. 2. Hysteresis loops for as-spun $[Nd_{0.8}(Dy_{0.5}Y_{0.5})_{0.2}]_{10}Fe_{84-w}B_6Zr_w$ alloys with various Zr additions.

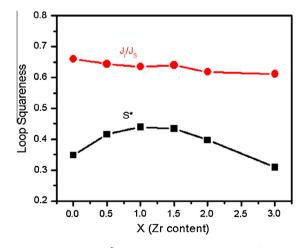


Fig. 3. The squareness factor S^* , defined by the ratio of the reverse field required to reduce J by 10% from the remanence to H_{cj} , and remanence ratio J_r/J_s for the alloys with various Zr doping.

magnetic polarization J_s decrease, as a result, the remanence J_r decreased with increasing Zr addition. On the other hand, with x increases from 0 to 2.0, the coercivity H_{cj} increases. For example,

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